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FOREIGN TECHNOLOGY DIVISION

SYNTHESIS OF ORGANIC COMPOUNDS

(Selected Articles)

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Б б	*Б б*	B, b	С с	*С с*	S, s
В в	*В в*	V, v	Т т	*Т т*	T, t
Г г	*Г г*	G, g	У у	*У у*	U, u
Д д	*Д д*	D, d	Ф ф	*Ф ф*	F, f
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К к	*К к*	K, k	Ъ ъ	*Ъ ъ*	"
Л л	*Л л*	L, l	Ы ы	*Ы ы*	Y, y
М м	*М м*	M, m	Ь ь	*Ь ь*	'
Н н	*Н н*	N, n	Э э	*Э э*	E, e
О о	*О о*	O, o	Ю ю	*Ю ю*	Yu, yu
П п	*П п*	P, p	Я я	*Я я*	Ya, ya

*ye initially, after vowels, and after ъ, ь; e elsewhere.
When written as ё in Russian, transliterate as yё or ё.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	$\sinh^{-1}$
cos	cos	ch	cosh	arc ch	$\cosh^{-1}$
tg	tan	th	tanh	arc th	$\tanh^{-1}$
ctg	cot	cth	coth	arc cth	$\coth^{-1}$
sec	sec	sch	sech	arc sch	sech^{-1}
cosec	csc	csch	csch	arc csch	csch^{-1}

Russian	English
rot	curl
lg	log

GRAPHICS DISCLAIMER

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SYNTHESES OF ORGANIC COMPOUNDS

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3. ALLYL-ORTHOTITANATE

$(CH_2=CH-CH_2O)_4Ti$

$C_{12}H_{20}O_4Ti$ Mol. wt. 276.19

O. V. Nogina, R. Kh. Freydlina

$$4CH_2=CHCH_2OH+TiCl_4+4NH_3 \rightarrow (CH_2+CHCH_2O)_4Ti+4NH_4Cl$$

Allyl ester of orthotitanium acid is obtained for the first time [1].

The proposed method is based on the reaction of allyl alcohol with titanium tetrachloride in the presence of ammonia in the medium of benzene.

DESCRIPTION OF SYNTHESIS

Synthesis is realized in essence as described for ethyl-orthotitanate (see pg. 164).

Into a three-necked round-bottomed flask with a capacity of 500 ml equipped with dropping funnel, reflux condenser and tube for the introduction of ammonia, are placed 102 g (1.75 g-mols) of thoroughly dried out allyl alcohol, which boils at 96-97° and 250 ml of absolute benzene (see note). Cooler and dropping funnel are connected with the drying tubes. From dropping funnel during cooling of reaction flask by ice water gradually during 30-40 min. are added 60 g (0.32 g-mols) of titanium tetrachloride, after which reaction mixture is saturated by dry ammonia before the achievement of increase in weight not less than 25 g (1.46 g-mols). Is filtered out the fallen ammonium chloride. Is distilled the residue after the distillation of benzene and excess allyl alcohol in the vacuum. Yield of allyl-orthotitanate is 36-40 g (41-46% of the theoretical). Boiling point is 141-142° at 1 mm.

All operations pointed out above are conducted without the access of moisture of air as this was described in the synthesis of ethylorthotitanate (pg. 164).

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PROPERTIES

Allyl-orthotitanate is colorless viscous liquid, boiling point 141-142° at 1 mm; it is hygroscopic and easily hydrolized.

NOTE

Synthesis can be carried out in the absence of benzene in the medium of allyl alcohol, taken with the excess.

REFERENCES

1. O. V. Nogina, R. Kh. Freydlina, A. N. Nesmeyanov. Bull. AN USSR, section of chem. sciences, 1950, 327.

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58. Ethylorthotitanate

$(C_2H_5O)_4Ti$

$C_8H_{20}O_4Ti$ Mol. wt. 228.14

O. V. Nogina, A. B. Belyavskiy

$$4C_2H_5OH+TiCl_4+4NH_3 \rightarrow (C_2H_4O)_4Ti+4NH_4Cl$$

Alkyl-orthotitanates can be obtained by the action of titanium tetrachloride on appropriate alcoholates of sodium [1]. According to short data of some patents [2] alkyl-orthotitanates are obtained under the effect of titanium tetrachloride on alcohols in the presence of amines. However, according to Speer [3], obtaining orthoesters by the last way is impossible.

The proposed method is based on the reaction of titanium tetrachloride with ethyl alcohol in the presence of ammonia and is characterized by the high yield of product [4].

DESCRIPTION OF SYNTHESIS

Into the three-necked round-bottomed flask with capacity of 1500 ml, equipped with mixer with the mercury seal, dropping funnel and reflux condenser, connected with the drying tube, filled with phosphorous pentoxide, are placed 600 ml of absolute ethyl alcohol. During cooling of flask and mixing gradually they add from dropping funnel, connected with the chlorocalcium tube 142 g (0.75 g-mols), titanium tetrachloride. After this, dropping funnel is replaced by gas-intake tube and reaction mixture during the cooling on the ice bath and the mixing is saturated by dry ammonia before the achievement of the increase in weight of the reaction mass of 51 g (3 g-mols). Ammonia is dried by its passing through three columns, filled with the pieces of solid alkali. Further reaction mass rapidly is transferred to the funnel with the glass filter.

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They filter out the precipitated ammonium chloride. Filtration is produced without the access of moisture of air, for which they unite funnel with the drying tube, another drying tube is placed between the bottle for the suction and the aspirator. From the solution they distill alcohol, in this case settles the white precipitate, which they again filter out, as it is described above. After the final distillation of the residues of ethyl alcohol (see note) product they distill in the vacuum.

Boiling point of ethylorthotitanate of 130-132° with 2 mm. Yield 115-120 g (67-70% of the theoretical).

The operations of distillation conduct without the access of moisture of air, for which they unite capillary for the introduction of air into the distillation flask with the system of the drying tubes. Air induction into the instrument at the end of distillation is produced through the system of driers indicated.

PROPERTIES

Ethylorthotitanate is colorless liquid with the weak fruit odor. It is hygroscopic and easily will be hydrolized with moisture of air. Thermally very struts it is distilled at the atmospheric pressure without the decomposition at 236-237°.

NOTE

Distilled alcohol can be without further drying or cleaning newly used for the synthesis of ethylorthotitanate.

REFERENCES

1. F. Bischoff, H. Adkins. J. Am. Chem. Soc., 1924, 46, 256; D. W. MacCorquodale, H. Adkins. J. Am. Chem. Soc., 1928, 50, 1938.

2. French pat. 818570; Zbl., 1937, II, 4102; Dutch. pat. 44107; Zbl., 1939, I, 1856.

3. R. J. Speer. J. Org. Chem., 1949, 14, 655.

4. O. V. Nogina, R. Kh. Freydlina, A. N. Nesmeyanov. Bull. AN USSR, section of chem. sciences, 1950, 327.

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